भारतीय मानक
Indian Standard

IS 1448 [P : 95] : 2019

पेट्रोलियम एवं इसके उत्पादों की परीक्षण पद्धतियाँ

[पी : 95]

चिकनाई तेल की डीमुल्सिबिलिटी गुणधर्मों का निर्धारण

(पहला पुनरीक्षण)

Methods of Test for Petroleum and its Products

[P : 95]

Determination of Demulsibility Characteristics of Lubricating Oils

(First Revision)

ICS 75.160.20

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Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard [P : 95] (First Revision) was adopted by the Bureau of Indian Standards on recommendation of the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1980 which was an adaptation of IP 19. In this revision, reagents required and procedure for cleaning of apparatus are included.

Considerable assistance was derived from IP 19: 2012 'Determination of demulsibility characteristics of lubricating oil'.

When a petroleum oil is mixed with water, it usually separates upon standing. The presence of minute contaminating materials decreases the rate of separation and may cause formation of stable emulsions. For liquids, an emulsion is the suspension of small globules of one in the other.

For some special purposes, it is desirable that an oil form a relatively stable emulsion with water. However, for most other purposes, it is important that the oil separates rapidly and completely from the water. This usually happens when steam or water leaks into the oil system. Various standards are available for testing the quality of oils in the respect. One such standard is the "Demulsification Number" which is also known as the "Steam Emulsion Number" and the unit of measurement is 'sec'.

In this method, steam is passed into the sample under definitely prescribed conditions. The time in seconds that is, required for complete separation of the oil and condensed steam is the steam emulsion number of the sample.

The test is sensitive to certain foreign materials but cannot be used to determine cleanliness because there materials have widely variable effects on the Steam Emulsion Number.

The test for steam emulsion is important for turbine oils because they frequently come in contact with water or steam as the oil flows through the circulation system. If the oil and water form a permanent emulsion, it will reduce the effectiveness of lubrication and contribute to the formation of water and sludge. Such emulsions may be due to impurities dissolved in the oil, or to the condition of the system into which the oil is introduced or to contamination such as fly ash from the air. When a new oil is put into an installation, it is important that the circulating system be clean, free of residual rust preventives, sludge, or other coatings from previous services.

Steam emulsion numbers determined on turbine oils inhibited against oxidation and rust formation are unreliable as shown by correlation with results obtained in service. The method for steam emulsion number is used only occasionally for other oils. Its value depends upon the type of oil and, at best, its correlation with service is no better than for turbine oils.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 95]

DETERMINATION OF DEMULSIBILITY CHARACTERISTICS OF LUBRICATING OILS

(First Revision)

1 SCOPE

1.1 This Indian Standard [P : 95] describes the method of the measure of ability of the oil to separate from an emulsion. It is commonly applied to turbine oils, but it may be used for other lubricating oils. The test is commonly applied to used turbine oils but since it is sensitive to ageing and contamination of the oil, precision will be lower than that stated.

1.2 Oils containing inhibitors may give much higher results than the corresponding uninhibited oils and the precision may be less satisfactory.

2 REFERENCES

The following standard contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

International Standard	*Title*
ISO 3696 : 1987	Water for analytical laboratory use — Specification and test methods

3 TERMINOLOGY

For the purpose of this standard, the following definition shall apply.

3.1 Demulsification Number — The number of seconds required for an oil to separate when it is emulsified and separated under specified conditions.

4 OUTLINE OF THE METHOD

Twenty milliliters of oil is emulsified with steam at about 90°C. The emulsion is then placed in a bath at about 94°C and the time for 20 ml of oil to separate is recorded.

5 APPARATUS

5.1 The apparatus consists of the following parts and is as shown in Fig. 1.

5.1.1 *Steam Generator*

Of metal or glass, and if of metal fitted with water-gauge.

5.1.2 *Separating and Emulsifying Baths*

Of glass, and provided with wooden or metal covers to hold the oil container and thermometers vertically in the bath.

5.1.3 *Heat Sources*

A suitable burner or electric hot plate for the steam generator and any convenient means, such as an auxiliary steam line, for the separating bath.

5.1.4 *Oil Container Tube*

5.1.5 *Steam Piping*

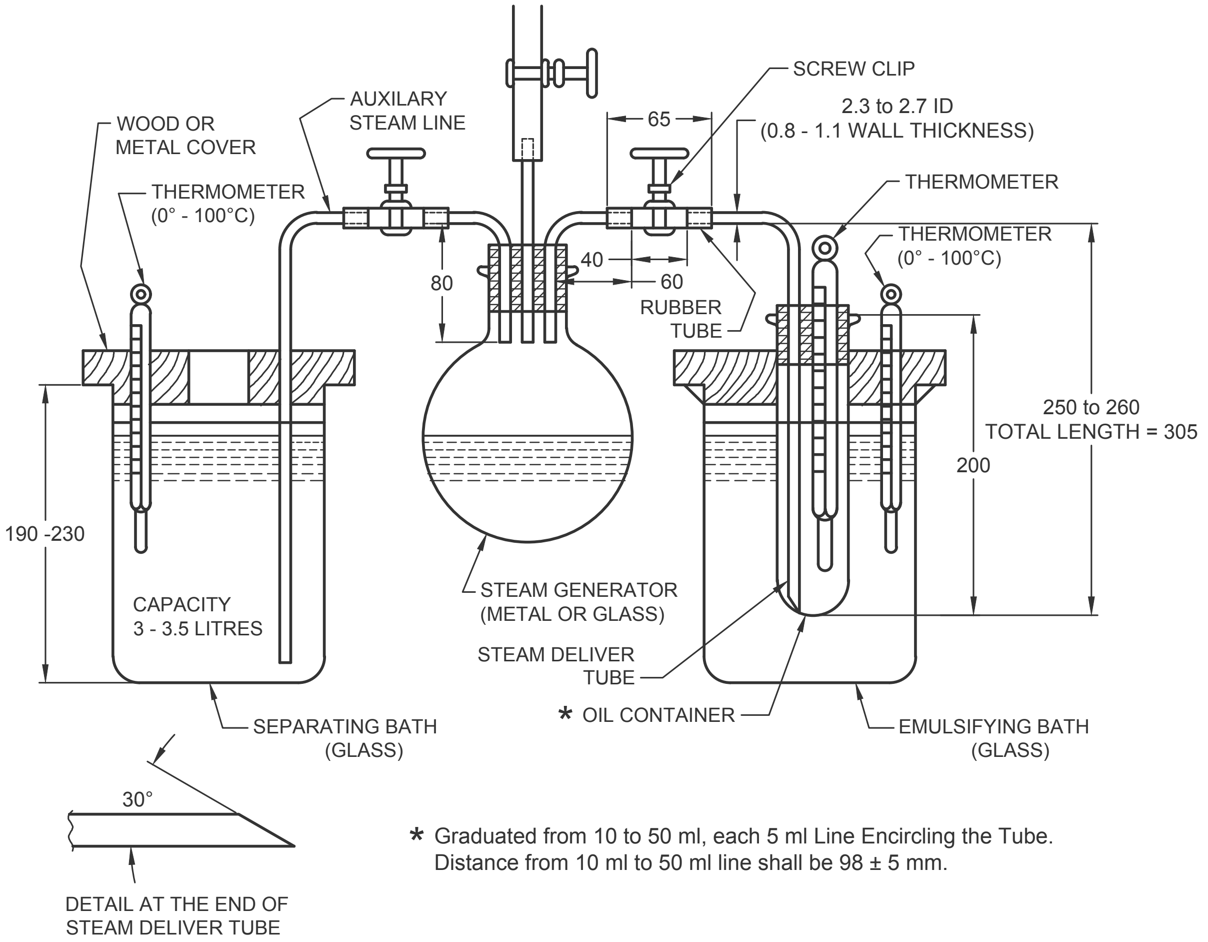
5.1.6 *Thermometers for the Separating and Emulsifying Baths*

Of suitable range, that is, 0 to 100°C.

5.1.7 *Thermometer for Oil Container*

Conforming to the following requirements:

Range	-1°C to +105°C
Graduation at each	0.5°C
Immersion	Total
Stem diameter	5.5 to 8.0 mm
Bulb shape	Cylindrical
Bulb diameter	Not greater than stem
Length of graduated portion, *Min*	170 mm
Longer lines at each	1°C and 5°C
Figured at each	5°C
Expansion chamber	Required
Top finish	Ring
Scale error, *Max*	± 0.5°C



* Graduated from 10 to 50 ml, each 5 ml Line Encircling the Tube. Distance from 10 ml to 50 ml line shall be 98 ± 5 mm.

All dimensions in millimetres

Fig. 1 Apparatus for Determination of Demulsification Number

6 REAGENTS

Use only reagents of recognized analytical grade.

6.1 Ammonium Peroxydisulfate — Store in a cold, dry place.

6.2 Sulfuric Acid Concentrated — Minimum purity 98 percent (v/v).

6.3 Ammonium Peroxydisulfate Cleaning Solution 8 g/l — Dissolve 8 g of ammonium peroxydisulfate (**5.1**) in the minimum amount of water and transfer whilst stirring to a 2 litre beaker containing 1 litre of concentrated sulfuric acid (**5.2**).

6.4 De-ionized Water — unless otherwise specified, meeting the requirements for grade 3 of ISO 3696.

7 PRECAUTIONS

The results obtained by this method are readily affected by the presence of traces of impurities in the sample and in consequence, it is most important to avoid contamination of the sample. Take precautions to exclude light from the sample until it can be tested.

8 PREPARATION OF APPARATUS

8.1 Fill the steam generator half-full of water, adding some broken pumice stone for the promotion of even ebullition, and assemble the apparatus as shown in Fig. 1. Take care to exclude from the steam generator any foreign materials which would cause contamination of the steam, and make sure that the steam-piping is clean. Fill each bath with 3 000 ± 60 ml of water.

8.2 Boil the water in the steam generator; adjust the temperature of the water in the emulsifying bath to 19 to 26°C and in the separating bath to 93 to 95°C.

8.3 Fill the oil container tube with the ammonium peroxydisulfate solution and allow to stand overnight in a fume cupboard. Remove the cleaning solution and rinse several times with water.

9 PROCEDURE

9.1 Ensure that all parts of the apparatus, which are exposed to the sample, are chemically clean, since this test is very susceptible to contaminants. Measure

20 ml of the sample into the oil container and place the latter in the holder of the emulsifying bath. Place a cork with two openings, with the thermometer in one, in the mouth of the oil container, adjusting the thermometer so that the bottom of the bulb is 20 to 25 mm from the bottom of the tube.

9.2 Carry out the test in duplicate.

9.3 Pass steam through the steam-delivery tube until the condensate disappears, and then disconnect it from the rubber tubing and insert it through the second opening in the cork until the end of the tube touches the center of the bottom of the oil container. The fit between the steam-delivery tube and the cork should be loose. Re-connect the rubber tubing to the steam-delivery tube and admit steam at such a rate that the temperature of the oil, as indicated by the thermometer in the oil container, rises to 88 to 91°C. The usual time for the temperature of the oil to reach this point is 45 to 75 s depending on its nature. Control the temperature by regulating the screw clips on steam pipe and steam exhaust, making sure that the steam supply is sufficient at all times to cause a generous discharge from the exhaust. Continue passing steam until the volume of the condensed water and the oil in the oil container is 40 ± 3 ml, making appropriate allowance for the volume of steam, the steam-delivery tube and the thermometer; this volume is approximately 4 ml. The time for this operation should be 4 to 6.5 min. If the time is less than 4 min, reject the test as the steam is probably wet, or the steam-delivery tube was completely steamed out.

9.4 Disconnect the steam-delivery tube from the rubber tubing and immediately start a stop-watch. Without delay, transfer the oil container to the separating bath; it is extremely important that the separating bath be maintained at 93 to 95°C. Remove the thermometer and steam-delivery tube from the oil container (*see* Note). After every 30 s record the volume of the separated oil layer, making no differentiation between the clear and turbid oil. Where the interface between more or less clear oil and the emulsion is not a clear, straight, horizontal line, estimate the position of such a line to the nearest 0.5 ml, mid-way through the more or less clear oil bubbles. Consider any lacy emulsion as part of the water layer. Record the time in seconds at the end of the 30 s interval during which 20 ml of oil separates. If 20 ml of oil has not separated in 20 min, discontinue the test and record the volume of oil separated.

NOTE — The oil container shall not be withdrawn from the separating bath during the period of examination.

10 CALCULATION AND REPORTING

10.1 Calculate the mean of the results of the duplicate determinations. If these results differ from the mean by more than 15 s or by more than 10 percent of the mean, if this is greater, make a third test.

10.2 Report the mean result to the nearest 15 s as the demulsification number in secods. If 20 ml of oil has not separated in 20 min, report the demulsification number as 1 200 + (number of ml separated).

11 PRECISION

Results of duplicate tests should not differ by more than the following amounts:

	Repeatability	*Reproducibility*
Unused oil	30 s or 20 percent of mean, whichever is greater	60 s or 30 percent of mean whichever is greater

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Amendments Issued Since Publication

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